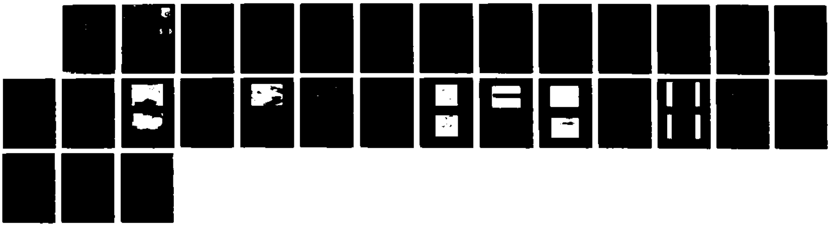
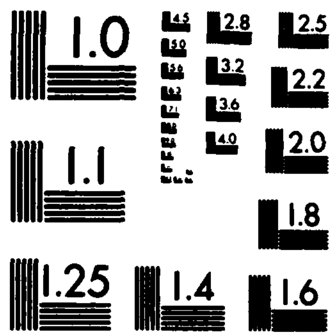
AD-A185 014 DEVELOPMENT OF ENCAPSULATED DYE FOR SURFACE IMPACT 1/1
DAMAGE INDICATOR SYSTEM(U) AMES LAB IA G LIGHT ET AL.
SEP 87 AFWAL-TR-87-4080 MIPR-FY1457-85-N-5021
UNCLASSIFIED F/G 11/4 NL





MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

AFWAL-TR-87-4080

DEVELOPMENT OF ENCAPSULATED DYE FOR SURFACE IMPACT DAMAGE INDICATOR SYSTEM

Dr. D. O. Thompson
Ames Laboratory
Iowa State University
Ames, Iowa 50011

Final Report for Period July 85 - December 86

September 1987

Approved for public release; distribution unlimited.

MATERIALS LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433-6533

NOTICE

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely Government-related procurement, the United States Government incurs no responsibility or any obligation whatsoever. The fact that the Government may have formulated or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication, or otherwise in any manner construed, as licensing the holder, or any other person or corporation; or as conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

This report has been reviewed by the Office of Public Affairs (ASD/PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

Charles F. Buynak

CHARLES F. BUYNAK
Nondestructive Evaluation Branch
Metals and Ceramics Division

FOR THE COMMANDER

D. M. Forney, Jr.

D. M. FORNEY, JR., Chief
Nondestructive ~~Evaluation~~ Branch
Metals and Ceramics Division

QUALITY INSPECTED 2

Accesion For	
NTIS CRA&I	☑
DTIC TAB	☐
Unannounced	☐
Justification	
By	
Distribution /	
Availability Codes	
Dist	Avail and / or Special
A-1	

If your address has changed, if you wish to be removed from our mailing list, or if the addressee is no longer employed by your organization please notify AFWAL/MLLP, Wright-Patterson AFB, OH 45433-6533 to help us maintain a current mailing list.

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

87 9 18 105

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE

A185014

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED	1b. RESTRICTIVE MARKINGS
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited.
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE	
4. PERFORMING ORGANIZATION REPORT NUMBER(S)	5. MONITORING ORGANIZATION REPORT NUMBER(S) AFWAL-TR-87-4080
6a. NAME OF PERFORMING ORGANIZATION Southwest Research Institute 6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION Materials Laboratory (AFWAL/MLLP) AF Wright Aeronautical Laboratories (AFSC)
6c. ADDRESS (City, State, and ZIP Code) 6220 Culebra Road PO Drawer 28510 San Antonio, Texas 78284	7b. ADDRESS (City, State, and ZIP Code) AFWAL/MLLP Wright-Patterson Air Force Base OH 45433-6533
8a. NAME OF FUNDING/SPONSORING ORGANIZATION Department of Energy 8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER MIPR FY1457-85-N-5021 MIPR FY1457-86-N-5009
8c. ADDRESS (City, State, and ZIP Code) Chicago Operations Office (Attn: J. Mahoney) 9800 Cass Avenue Argonne IL 60439-4802	10. SOURCE OF FUNDING NUMBERS PROGRAM ELEMENT NO. 62102F; PROJECT NO. 2418; TASK NO. 06; WORK UNIT ACCESSION NO. 06

11. TITLE (Include Security Classification)

Development of Encapsulated Dye for Surface Impact Damage Indicator System

12. PERSONAL AUTHOR(S) Glenn Light, Ph. D.

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
Final	FROM 7/85 TO 12/86	September 1987	28

16. SUPPLEMENTARY NOTATION

17. COSATI CODES

FIELD	GROUP	SUB-GROUP
11	04	
11	03	

18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
Composites, Impact, NDE, Encapsulation, Ultrasonics, Microcapsules, Polyurethane, Paint, Dye

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

The results of two phases of a MIPR program to develop a rapid scan method of detecting the occurrence of foreign-object-damage (FOD) to composite structural components are reported. The Nondestructive Evaluation (NDE) technique is based on an encapsulated dye system within the paint to detect and partially quantify FOD to composite structures. The major accomplishments of this effort were the encapsulation, microencapsule incorporation into the USAF polyurethane paint, and initial correlation study of impact damage to impact coating indication. It is the conclusion of the work reported here that the encapsulated damage detection system offers a feasible rapid scan inspection system for carbon-epoxy composites that with further development could play a significant role in the NDE inspection of composite airframe structures.

Described are

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT ☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS	21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED
22a. NAME OF RESPONSIBLE INDIVIDUAL Charles F. Buynak	22b. TELEPHONE (Include Area Code) (513) 255-5561 22c. OFFICE SYMBOL AFWAL/MLLP

DD FORM 1473, 84 MAR — 83 APR edition may be used until exhausted. All other editions are obsolete.

SECURITY CLASSIFICATION OF THIS PAGE
UNCLASSIFIED

EXECUTIVE SUMMARY

Southwest Research Institute was funded by Ames Laboratory under Contract No. SC-86-111, entitled "Development of Encapsulated Dye for Surface Impact Damage Indicator System, Phase II," to improve upon the impact-indicative coating technology that was shown to be feasible in the previous Ames Laboratory Contract No. SC-85-091, entitled "Development of Encapsulated Dye for Surface Impact Damage Indicator System, Phase I." The major goals of this project were to:

1. Refine the microcapsule formulation to be compatible with MIL-C-83286 paint.

2. Fabricate composite panels from isotropic graphite epoxy for use during testing of the impact coating.

3. Conduct impact damage testing to determine the level of impact that causes damage.

4. Correlate the color of the dye to the impact levels so that different levels of impact could be differentiated.

5. Demonstrate the capability of an encapsulated dye system to indicate the location and intensity range of impact damage to composite structures.

The coating developed under this contract was compatible with the MIL-C-83286 paint used by the Air Force. In order to achieve compatibility with the Air Force paint, it was necessary to encapsulate both the dye and developer in separate capsules. When the coating was subjected to impacts, the capsules were broken by the pressure of the impact and a color was developed in the coating at the point of impact.

Impact tests conducted on three different thicknesses of graphite epoxy composite showed that impacts in the range of 5 ftlbs would cause internal matrix cracking and delamination without leaving a surface indication. Therefore, it was necessary to have an impact coating that would produce color indications for impacts greater than 5 ftlbs. In order to achieve breakage of the capsules at this impact level, the capsules had to be in the 5 to 10 micron diameter size range. Coatings were developed that clearly indicated impacts greater than 1 ftlb and impacts greater than 40 ftlbs. However, the goal of developing a coating that would indicate impacts greater than 5 ftlbs and show no indication for impacts less than 5 ftlbs was not achieved.

In addition, spray application methods were tested with the result that no damage to the microcapsules or clogging of the spray apparatus occurred. This indicated that the impact-sensitive coating has field application potential. Additional studies need to be conducted to optimize the coating for the proper impact level, to evaluate shelf life problems, and to determine effects of environment such as temperature range, humidity, and moisture.

TABLE OF CONTENTS

LIST OF FIGURES

INTRODUCTION

Background

Composite structure on aircraft is exposed to impact damage while on the ground, during taxi and takeoff, and during flight. Impacts can occur as a result of dropped tools, flying runway debris, and hailstorms. Research over the last few years has shown that impacts of a sufficient level can cause internal matrix cracking and delamination without leaving any physical evidence of the damage on the impacted surface (1). This internal damage can lead to loss of structural strength and ultimate failure of the composite component.

Based upon this information and as a result of previous successful impact indication programs (2,3,4) conducted at SwRI, SwRI was funded by Ames Laboratory to develop an impact coating that could be applied to the surface of the composite structure and that would visibly indicate impacts of a certain level. The initial feasibility of developing a coating that would indicate impact was successfully demonstrated under Contract No. SC-85-091.

Once the feasibility of developing an impact-sensitive coating was demonstrated, additional parameters of the coating had to be evaluated and improved to ultimately make the impact coating useful in the field. One of the major parameters was the chemical compatibility of the impact coating with the MIL-C-83286 paint used by the Air Force. In addition, at the beginning of the project, it was still unclear what level of impact caused internal damage to the composite. Finally, it was desired to develop an impact coating that would not only indicate the occurrence of an impact, but also give some estimate of the range of impact. Based upon these considerations, Phase II of the project was funded by Ames Laboratory.

Phase II Work Scope

During this project, the work to be acomplished was divided into five tasks. The objectives of these tasks were as follows:

Task 1. Refine microcapsule formulation to be compatible with MIL-C-83286 paint. Include parameters such as paint solvent resistance, capsule size, shelf life, and dye/activator compatibility.

Task 2. Fabricate three thicknesses of composite panels from isotropic graphite epoxy.

Task 3. Conduct impact damage testing on these panels to determine levels of impact that cause damage.

Task 4. Correlate dye color to impact loads. Provide a correlation between the indication produced by the impact-sensitive coating and the level of impact damage that occurs.

Task 5. Develop encapsulated dye surface impact damage indicator systems. Demonstrate the capability of an encapsulated dye system to indicate the location and intensity range of impact damage to composite structures.

DISCUSSION OF EXPERIMENTS AND RESULTS

The work conducted under each task is discussed in detail in the following paragraphs.

Task 1. Refinement of Microcapsule Formulation to be Compatible with MIL-C-83286 Paint

The purpose of this task was to evaluate coating parameters such as paint solvent resistance, proper capsule size, shelf life, and dye/activator compatibility. Studies during this task were concentrated on the development of two types of impact indicator systems; namely, visual color on impact, and ultraviolet (UV) fluorescence on impact. The system being investigated for visual color employs the use of a dye precursor (crystal violet lactone or CVL) which develops a dark-blue color in the presence of an activator (HJR 4002 or 4023 modified phenolic resin). The CVL or resin is dissolved in a water-immersible solvent and encapsulated as a solution. UV fluorescent materials investigated to date include CVL, carbazole, and anthracene. Table 1 lists the encapsulation experiments made during this task. Most of the microcapsules prepared during the program have been in the 1 to 10 micron diameter size range.

Table 2 gives a list of the results of impact tests on sample panels coated with different encapsulation formulations mixed in the two-part polyurethane paint. A primary consideration in the choice of materials to be utilized in the encapsulation program was the melting points and boiling points. The desired temperature characteristics are a melting point equal to -63 degrees C and a boiling point of +203 degrees C. This would enable the coating to withstand the expected temperature environment of an aircraft.

One coated specimen that had been impacted at various levels of impact was exposed to temperature extremes of -40 and +180 degrees Centigrade. No noticeable change in the coating was observed.

1(a) Visual Indicators

Early work on the visual impact indicator system involved the encapsulation of CVL (dye) for use in the two-part polyurethane paint which contained the dispersed activator resin. The CVL capsules and activator resin gave good color intensity when mixed together and crushed, but gave no color when mixed in the polyurethane paint and impacted. It was found that the isocyanate portion of the paint catalyst was reacting with the activator resin (developer), thereby destroying its usefulness as a color developer. To overcome this problem, we experimented with a reverse concept, i.e., to encapsulate the activator instead of the dye. Therefore, efforts since then have been directed toward encapsulation of the activator resin in order to protect it from the isocyanate in the paint system. Experiments (called "runs") WP-63 and WP-93 are examples of resin capsules which retain their activity in the paint and give color indication when impacted (see Table 2). In the cases where resin (activator encapsulated) microcapsules were used, the CVL was dissolved in the paint. The concentration of the microcapsule components in the paint is in the range of 4 to 12 weight percent of the uncured paint components.

TABLE I. MICROENCAPSULATION RUNS

Run No.	Shell System	Fill Material	Encapsulation Method	Remarks
WP-63	Gelatin-Gum Arabic + Sodium Trimethyl Silanolate	30% HRJ 4002 Resin in diphenyl methane (DAM) + L-31 reactive silicone fluid	Coacervation	Good capsules formed. Product spray dried using a Buchi 190 mini-spray dryer. Size = 1-10μ. Payload = ~55%
WP-70	Gelatin-Gum Arabic	Crystal Violet Lacetone (CVL) saturated in Diisopropyl Benzene (DIPB)	Coacervation	Capsules formed but fused together on heating at 70°C. Payload = ~ 60%
WP-71	Repeat WP-70	W/O heat treatment		Capsules formed and collected. No color when crushed in presence of 4002 resin actuator.
WP-72	Gelatin	CVL saturated in DIPB	Coacervation	Capsules prepared and collected. Payload = ~75%
WP-73	Gelatin + TiO_2	Fluorescein saturated in DIPB	Coacervation	Collected capsules fluoresce before crushing. Payload = ~70%
WP-74	Gelatin-Gum Arabic	Saturated 4002 resin in DIPB	Coacervation	Capsules formed and collected. No color indication when crushed with CVL. Payload = ~60%
WP-75	Gelatin-Gum Arabic	20% 4002 resin in 15/85 DPM/DIPB	Coacervation	Treat collected product with aminopropyl trimethoxy silane. Payload = ~71%
WP-76	Gelatin + Carbon Black	CVL saturated in DIPB	Cocervation	Collected product fluoresces under UV light. Payload = ~ 75%
WP-77	Gelatin + Carbon Black	CVL saturated in DIPB	Coacervation	Carbon black is added at different stage than in WP-76. Product fluoresces less than WP-76 under UV light. Payload = ~75%
WP-79	Gelatin + Carbon Black	2% CVL in 85/15 DIPB/DPM	Coacervation	Product fluoresces under UV light. Payload = ~71%

TABLE 1 (Continued)

Run No.	Shell System	Fill Material	Encapsulation Method	Remarks
WP-82	Polyurea + Poly (vinyl alcohol) and Carbon Black	2% CVL in 85/15 DIPB/DPM	Interfacial polymerization	Good product collected. Shows some UV fluorescence when crushed. Payload = ~67%
WP-83	Same as WP-82	1% fluorescein in DIPB	Interfacial polymerization	Product fluoresces.
WP-85	Polyurea + Poly (vinyl alcohol) and TiO_2	2% CVL in Benzoflex 9-88 (B9-88)	Interfacial polymerization	Blue product results. CVL reacts with polyurea reactants. Payload = ~71%
WP-87	Gelatin-Gum Arabic + Sodium Methyl Silanolate	10% 4002 resin in B9-88 + L-32 reactive silicone fluid	Coacervation	Product collected. Payload = ~64%
WP-88	Gelatin + Sodium Methyl Silanolate	2% CVL in B9-88 + L-32 reactive silicone fluid	Coacervation	Product collected. Payload = ~ 83%
WP-89	Same as WP-88	10% 4002 resin in B9-88 + L-32 reactive *silicone fluid*	Coacervation	Product collected. Payload = ~81%
WP-93	Gelatin-Gum Arabic + Sodium Methyl Silanolate	33% 4023 resin in 50/50 M-xylene/B9-88 + L-31 reactive silicone fluid	Coacervation	Capsules formed and collected. Payload = ~ 72%
WP-95	Polyurea-poly (vinyl alcohol)	Anthracene saturated in 10/1 BI-88/M-xylene	Interfacial polymerization	Product collected. No UV indication in impacted paint system. Payload = ~77%
WP-97	Same as WP-95	Carbazole saturated in B9-88	Interfacial polymerization	Product collected. No UV indication in impacted paint system. Payload = ~87%
WP-98	Gelatin-Gum Arabic + Sodium Methyl Silanolate + Poly (vinyl alcohol)	4% CVL in B9-88 + L-32 silicone fluid	Coacervation	Product collected. Payload = ~71%
WP-100	Polyurea + Poly (vinyl alcohol)	4% CVL in 25/75 DIPB/ B9-88	Interfacial polymerization	Product collected. Crushed capsules show fluorescence; however, paint becomes fluorescent after adding capsules. Payload = ~ 89%

TABLE 1 (Continued)

Run No	Shell System	Fill Material	Encapsulation Method	Remarks
WP-101	Repeat WP-98 at higher payload			No product recovered.
WP-102	Gelatin-Gum Arabic + Sodium Methyl Silanolate	30% 4023 resin in DPM + L-31 silicone fluid	Coacervation	Product collected. Very slight color indication. Payload = ~65%
WP-107	Methyl Cellulose	30% 4023 resin in DPM	Spray drying	Product collected. 60% payload. Turns blue in paint CVL mixture before curing. Payload = ~60%
WP-110	Methyl Cellulose - Poly (vinyl alcohol)	30% 4023 resin in DPM	Spray drying	Product collected. 75% payload. Turns blue in paint. Payload = ~75%
WP-113	Gelatin-Gum Arabic + Sodium Trimethyl Silanolate	CVL saturated in 60/20/20 B9-88/m-xylene/DIP.B + L-31 reactive silicone fluid	Coacervation	Capsules turn blue in air after drying. Payload = ~71%
WP-115	Gelatin	CVL saturated in 2/1 DIP.B/B9-88	Coacervation	Product collected. No impact indication with WP-93. Payload = ~ 75%
WP-117	Gelatin-Gum Arabic	12% CVL in 1/1 DPM/B9-88	Coacervation	Product collected. Very little impact indication with WP-93. Payload = ~83%
WP-120	Gelatin-Gum Arabic	Saturated CVL in m-xylene	Coacervation	Product collected. Size 5-15μ, give impact indication with WP-63. Payload = ~86%
WP-122	Gelatin-Gum Arabic	HRJ 4002 resin saturated in 80/20 m-xylene/B9-88	Coacervation	Product collected. Some indication with WP-126 on heavy impact. Payload = ~86%
WP-123	Gelatin-Methyl Cellulose	4% copikem x IV in B9-88	Spray drying	Product collected. Turns orange in paint with WP-110 capsules. Payload = ~76%
WP-126	Gelatin-Gum Arabic	Saturated CVL in 1/1 m-xylene/B9-88	Coacervation	Some product collected. Some indication with WP-122 on heavy impact; good impact indication with WP-63 on heavy impact
WP 128	Gelatin-Gum Arabic + Sodium Trimethyl Silanolate	30% 4002 resin in 2/1 DPM/B9-88 + L-31 reactive silicone fluid	Coacervation	Product collected. No impact indication with WP-126. Payload = ~60%

TABLE 2. RESULTS OF IMPACT TEST ON PANELS COATED WITH IMPACT INDICATOR OF THE LISTED CONCENTRATION

Run Number	Concentration in Paint (Weight Percent)	Remarks
WP-85	6	Coated onto fiberglass panel. Produced no indication when impacted in range of 5-10 ftlbs.
WP-82	6	Coated onto fiberglass panel. Produced no indication when impacted in range of 5-10 ftlbs.
WP-83	6	Coated onto fiberglass panel. Produced no indication when impacted in range of 5-10 ftlbs.
WP-88	6	Coated onto fiberglass panel. Produced no indication when impacted in range of 5-10 ftlbs.
WP-87 + WP-88	12	Coated onto fiberglass panel. Produced no indication when impacted in range of 5-10 ftlbs.
WP-88 + WP-89	12	Coated onto fiberglass panel. Produced no indication when impacted in range of 5-10 ftlbs.
WP-87 + neat CVL*	9	Coated onto fiberglass panel. Produced no indication when impacted in range of 5-10 ftlbs.
WP-89 + neat CVL	9	Coated onto fiberglass panel. Produced no indication when impacted in range of 5-10 ftlbs.
WP-93 + neat CVL	4	Coated onto graphite epoxy panel. Blue indication upon impact.
WP-63 + neat CVL	7	Coated onto graphite epoxy panel. Indication on impact.
WP-63 + neat CVL in undercoat	7	Coated onto graphite epoxy panel. No indication on impact when the CVL is in pigmented coating and microcapsules are in clear overcoat.
WP-120 + WP-63	30	Coated onto graphite epoxy panel previously painted white. Good impact indication. Sensitive to impacts as small as 1 ftlb.
WP-120 + WP-63	20	Spray coated onto graphite epoxy panel mixed with titanium oxide pigmented paint. Heavy impact was required to give indication.
WP-120 + WP-93	25	Coated onto graphite epoxy panel. Only slight indication upon impact.
WP-122 + WP-126	30	Coated onto graphite epoxy panel. Some indication tion with heavy impact.
WP-126 + WP-128	30	Coated onto graphite epoxy panel. No impact indication.
WP-126 + WP-63	40	Coated onto graphite epoxy panel. Indication with heavy impact.

*Neat CVL means that the CVL is added directly to the paint and is not encapsulated.

During the shelf life test, a problem was found with the WP-63 and WP-93 capsule formulation when used with neat CVL. Although the above indication system using encapsulated activator resin and neat CVL in the polyurethane paint gave an indication upon impact (in the range of 5 ftlbs), it was found that the entire coating turned blue after a few days. This apparently is caused by the reaction of the CVL with paint components in the presence of light. In an attempt to prevent this color change from occurring, the CVL was encapsulated for use in the indicator coating (such as runs WP-120 and WP-126 in Table 1). The CVL capsules were then used in combination with activator resin capsules in the polyurethane paint as the impact indicator coating. Impact indication was obtained using coatings containing WP-120 plus WP-63 capsules and WP-126 plus WP-63 capsules (see entries in Table 1 and Table 2).

Solvents for the CVL and activator resin used during the program include diphenylmethane (DPM), diisopropylbenzene (DIPB), metaxylene, and Benzoflex 9-88 (B9-88, dipropylene glycol dibenzoate). These were used alone and/or in combination with each other (see Table 1 under fill material). The DPM is a particularly good solvent for the activator resin and was used to prepare the capsules (run WP-63) that gave the best indications of impact.

Preliminary spray coating trials with the capsules dispersed in polyurethane paint indicated that the microencapsulated coating system can be spray painted onto surfaces without plugging the nozzle or damaging the capsule. The capsules were dispersed in the paint with the aid of an ultrasonic probe and applied with a model 400 Badger spray gun.

1(b) Ultraviolet Fluorescent Indicators

Several materials have been evaluated as ultraviolet fluorescent indicators. However, no capsule formulation was developed that gave an indication upon impact. For example, capsules containing CVL (run WP-100 from Table 1) were prepared which produced UV fluorescence when crushed; but when placed in the polyurethane paint, the paint itself became UV fluorescent. This is believed to be caused by the permeation of the CVL in the paint solvents. It is anticipated that systems developed for the visible impact indicators will be applicable to the UV fluorescent capsules if the capsules can be coated to mask the fluorescence of materials prior to impact. The masking will be accomplished by including carbon black or titanium dioxide pigment in the capsule shell. However, no additional UV work was conducted during this task.

1(c) Different Color Indications

Both blue and red colored dyes were used during the project. The blue worked much better, but the red was useable. The ultimate concept was to put different color dyes in different strength capsules to correlate impact level to color. This was not completed in this program, but is certainly feasible.

Task 2. Fabrication and Inspection of Composite Panels from Isotropic Graphite Epoxy

All graphite epoxy composite panels were fabricated by the University of Dayton and received at SwRI during March 1986. The panels were made in three thicknesses, i.e., 8 ply, 16 ply, and 48 ply and were made from Hercules

IM-6 graphite fiber/3501-6 epoxy resin. A total of twelve panels of each thickness were fabricated in a 24-inch by 24-inch plate form. These panels were then cut into test specimens that were 5-3/4 inch by 5-3/4 inch. The University of Dayton performed density tests and the average parameter values for each type of composite panel are shown in Table 3.

TABLE 3. PHYSICAL PROPERTIES DATA

Panel	Density		Resin Content		Fiber Volume		Void Volume	
Type	g/cc	Sigma	% by Wt	Sigma	% by Vol	Sigma	% by Vol	Sigma
8 ply	1.57	0.03	26.12	1.14	66.90	1.97	0.77	0.7
16 ply	1.56	0.01	28.53	0.51	64.34	0.60	0.51	0.6
48 ply	1.58	0.04	26.11	6.29	67.54	7.54	0.23	0.4

The quasi-isotropic layup for the panels was as follows:

for the 8 ply (0/+45/-45/90)s
for the 16 ply (0/+45/-45/90/90/-45/+45/0)s
for the 48 ply (0/+45/-45/90/90/-45/+45/0/0/+45/-45/90)2s

These samples satisfy the need for composite test panels required under the scope of this program.

Task 3. Impact Damage Testing to Determine Levels of Impact That Cause Damage

The purpose of the impact testing was to simulate expected impact levels and impacts to which the composite structure could be subjected. This included such impacts as tools being dropped onto the composite, runway debris, and hail. A calibrated set of impactors which simulate the surface and levels of impact were designed. These include a hard, sharp object (end of a wrench or screwdriver), a hard, blunt object (hammer or other rounded object), and hailstones.

3(a) Impactors

The hard, blunt impactors used during this task were stainless steel balls ranging from 0.5 inch to 2.0 inches in diameter. These are shown in Figure 1. The hard sharp impactors were 2-inch diameter cylinders made of carbon steel. To achieve different masses, the lengths of the cylinders were varied. These cylinders are shown in Figure 2. The dimensions and weights are listed in Table 4.

These impactors were used to impact panels of the graphite epoxy composite. The test panels were 6 inches by 6 inches and were tested in both the supported and unsupported backing mode. For the drop tests, the supported mode consisted of laying the panel on a concrete slab and the unsupported mode consisted of placing the panel on a wood box frame with a box opening 5 inches

Figure 1. Photograph of the stainless steel balls used as hard blunt impactors during this program.

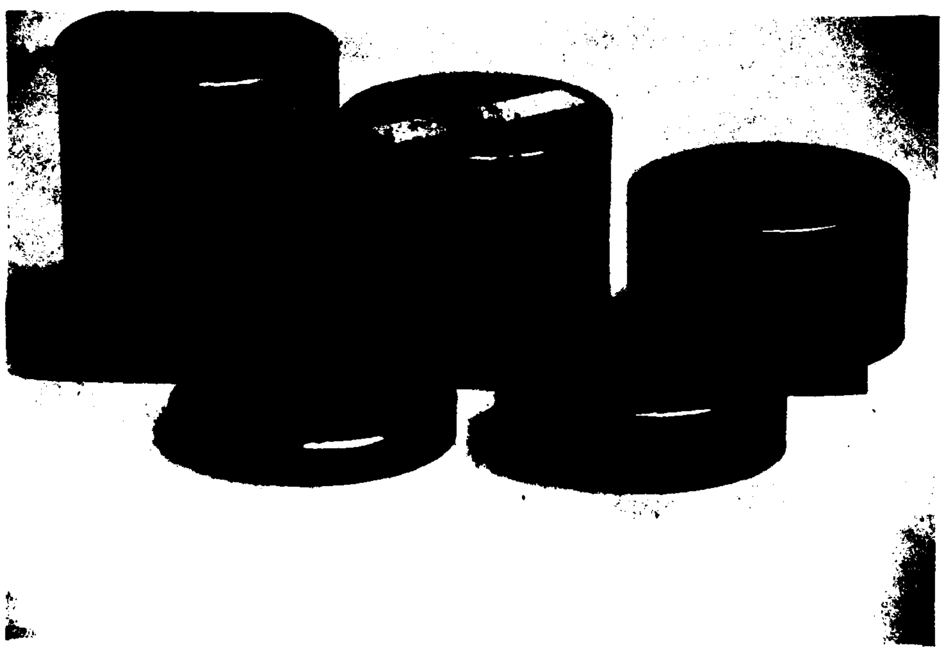


Figure 2. Photograph of the steel cylinders used as hard sharp impactors during this program.

TABLE 4. PHYSICAL PARAMETERS OF HARD BLUNT AND HARD SHARP IMPACTORS

Shape	Diameter (inch)	Length (inch)	Weight (lbs)
Sphere	0.562		0.026
Sphere	0.750		0.062
Sphere	0.935		0.12
Sphere	1.50		0.49
Sphere	1.87		0.96
Cylinder	2.0	0.296	0.25
Cylinder	2.0	0.575	0.50
Cylinder	2.0	1.13	1.00
Cylinder	2.0	1.68	1.50
Cylinder	2.0	2.25	2.00

by 5 inches. Tests have been conducted on all three composite panel thicknesses.

3(b) Impact Tests

Prior to the drop test (considered to be low velocity impacts), each panel to be impacted was inspected using a through-transmission ultrasonic C-scan technique. Theoretically, if damage such as cracks and delamination occurs, then the amount of ultrasonic energy transmitted through the area will decrease. Therefore, a means to determine composite damage using the C-scan is to indicate regions of the composite panel that absorb (or do not effectively transmit) the ultrasonic beam. The ultrasonic transducers used during the inspection were 1/2-inch diameter, 2.5 inch focal length in water, 5 MHz units. The panels were placed in a water bath and the C-scan performed as shown in Figure 3. After the impact the same system was used to again inspect each panel tested. Results of these tests are shown in Figures 4 and 5.

Figure 4 shows the C-scan results for panel 16-1-8 (a 16-ply panel) before and after impact by the hard blunt impactors (spheres). There were some indications on the panel prior to the impact. These indications were false and were due to water bubbles under the surface of the composite panel which prevented proper transmission of the ultrasonic beam. However, no major ultrasonic reflections appear. (Note that the linear indications shown in both C-scans are due to the holding apparatus used during the inspection.)

After impact in the unsupported mode, four distinct ultrasonic reflectors are observed. The two lower reflectors are due to impacts of 3 ftlbs while the two upper reflectors are due to 6 ftlbs of impact. Similar results were obtained from the drop tests of the hard sharp impactors.

Figure 5 shows the C-scan images for panel 16-1-10. The C-scan image before impact shows no ultrasonic reflectors. After the panel had been subjected to impacts of 3, 6, 9, and 12 ftlbs in the unsupported mode, C-scans were again obtained. These data are shown in the after impact case in Figure 5. Ultrasonic damage is observed for the 6 and 12 ftlb case, and not for the 3 and 9 ftlb cases. This does not seem logical. However, when the impacted region is studied it becomes clear that the hard sharp impactor hit on its edge in all

Figure 3. Photograph of the ultrasonic apparatus used to obtain C-scan images of the composite panels before and after impact.

cases except the 9 ftlb case, where it clearly hit flat and spread the impact over a 2-inch diameter. This caused less internal damage and thus produced no ultrasonic reflectors. Table 5 lists the panels used in the test, their thicknesses, the impactor type, the impact level, and results of ultrasonic tests.

3(c) Destructive Assay

After the test panels had been subjected to impact tests and ultrasonically inspected, they were destructively assayed. The results are discussed below.

The samples were visually inspected and photographed prior to destructive cutting. Some of the panels showed evidence of burnishing as well as cuts. Some cuts were as long as 1/2 inch and as deep as 1/16 inch. Several plates were destructively tested. These were 16-1-8, 16-1-10, 48-3-7, and 48-3-9. The C-scan data for these panels are shown in Figures 4 and 5. The ultrasonic C-scan data were used to determine cut lines since the C-scan data correlated well with the points of impact.

Photographs of two panels (16-1-8 and 16-1-10) are shown in Figures 6 and 7. The impactor for panel 16-1-8 was hard blunt (sphere) and the impactor for panel 16-1-10 was hard sharp (cylinder). Panel 16-1-8 showed no visible sign of damage due to impact, while panel 16-1-10 showed several cut regions. The impact levels for regions 1, 2, 3, and 4 on panel 16-1-8 were 3, 3, 6, and 6 ftlbs, respectively. The impact levels on panel 16-1-10 for regions 1, 2, 3, and 4 were 3, 6, 9, and 12 ftlbs.

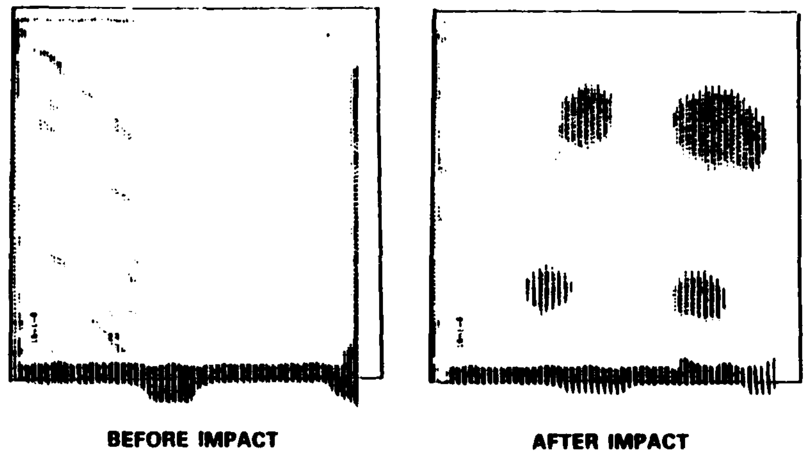


Figure 4. C-scan results for Panel 16-1-8 (a 16-ply panel) before and after impact by the hard blunt impactors (spheres). There are some indications on the panel prior to the impact due to water bubbles under the surface of the composite panel which prevented proper transmission of the ultrasonic beam. However, no major ultrasonic reflections appear. (Note that the linear indications shown in both C-scans are due to the holding apparatus used during the inspection.) After impact in the unsupported mode, four distinct ultrasonic reflectors are observed. The two lower reflectors are due to impacts of 3 ftlbs while the two upper reflectors are due to 6 ftlbs of impact.

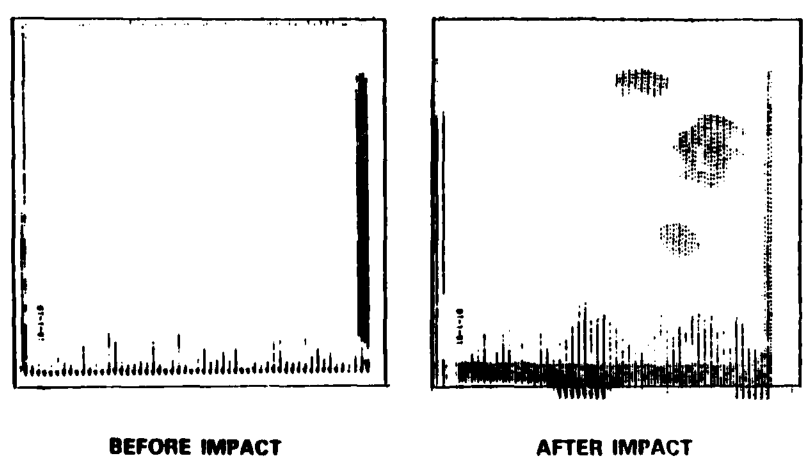


Figure 5. C-scan images for Panel 16-1-10 due to hard sharp body impacts. The C-scan image before impact shows no ultrasonic reflectors. After the panel has been subjected to impacts of 3, 6, 9, and 12 ftlbs in the unsupported mode, ultrasonic damage is observed for the 6 and 12 ftlb cases, and not for the 3 and 9 ftlb cases. The hard sharp impactor hit on its edge in all cases except the 9 ftlb case, where it clearly hit flat and spread the impact over a 2-inch diameter.

TABLE 5. PARAMETERS AND RESULTS OF IMPACT TESTS ON GRAPHITE EPOXY PANELS

Panel Number	Impactor Type*	Impact Level**	Support ***	Results of Impact
16-1-7	HB	3 & 6	S	C-scan showed indication for 3 and 6 ftlb impact levels
16-1-8	HB	3 & 6	U	C-scan showed indication for 3 and 6 ftlb impact levels
16-1-9	HS	3 to 12	S	C-scan showed indication for 6, 9, and 12 ftlb impact levels
16-1-10	HS	3 to 12	U	C-scan showed indication for 6 and 12 ftlb impact levels
48-3-7	HB	1 to 6	S	C-scan showed indication for 6 ftlb impact levels
48-3-8	HB	1 to 6	U	C-scan showed no indication
48-3-9	HS	3 to 12	S	C-scan showed indication for 6, 9, and 12 ftlb impact levels
48-3-10	HS	3 to 12	U	C-scan showed no correlatable indication

*HB and HS refer to hard blunt (sphere) and hard sharp (cylinder) impacts.
**Impact level is in ftlbs.
***S refers to supported and U refers to unsupported conditions for the plate.

Sections of the two panels were cut and inspected using a scanning electron microscope (SEM). The sections were cut through the middle of the damaged regions indicated by the ultrasonic C-scan. A typical SEM of the panel thickness is shown in Figure 8 for panel 16-1-8 (impact area 4). The damage pattern is typical. Matrix cracks which radiate from the center of impact are evident. Delaminations occur between many layers. It is important to note that for this panel there was no evidence of surface damage from a 6 ftlb, hard blunt (sphere) impact. Figures 9 and 10 show two regions of the panel wall at a higher magnification. The delamination and cracking is very clearly shown. Other panels showed similar results.

Based upon these results, it is clear that subsurface damage can occur to composite structures even though no outer visible damage occurs. This can occur with impact levels on the order of 6 ftlbs. This result proves the importance of successfully developing an impact-sensitive coating that can be placed on the surface of the composite to indicate occurrence of impacts.

3(d) Ice Pellet Impact Tests

In order to simulate effects of hailstone damage, ice pellet impact studies were conducted. The ice pellet impact tests were conducted using the SwRI air gun facility. Ten panels were impacted with ice pellets in an effort to study damage due to hail impact. The ice pellets used for the tests were 0.88 inch in diameter with length of 1, 2, and 3 inches. The exit velocity of the projectile was measured utilizing a time-of-flight photodetector system. The goal of this work was to generate various levels of impact at two velocity ranges; namely, 150 and 450 ft/sec. The panels impacted and the velocity and impact range obtained are given in Table 6.

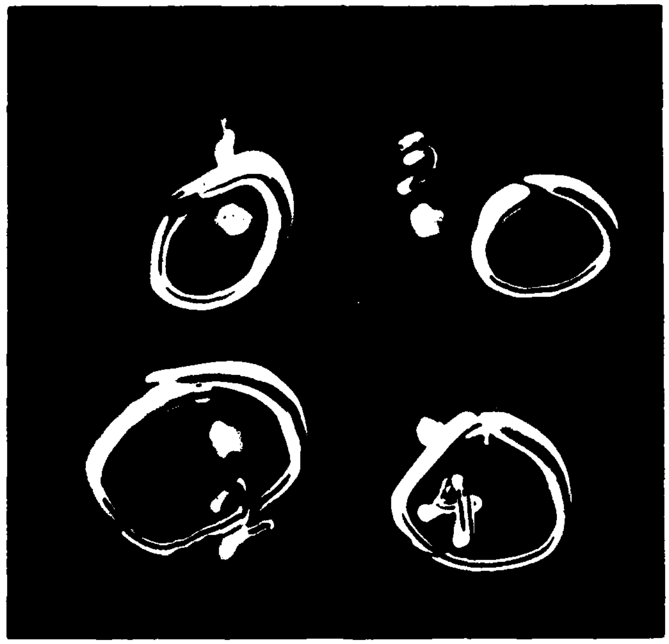


Figure 6. Photograph of the top surface of graphite epoxy sample No. 16-1-8 showing areas where impacts occurred.

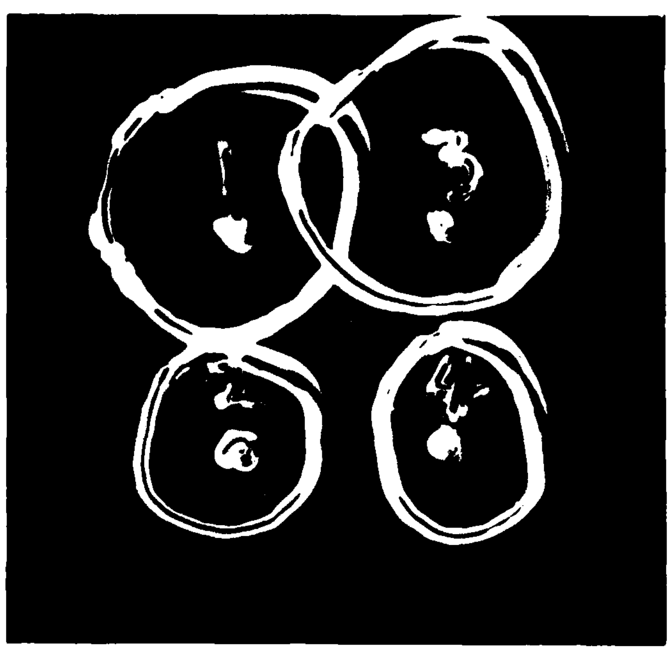


Figure 7. Photograph of the top surface of graphite epoxy sample No. 16-1-10 showing areas where impacts occurred.

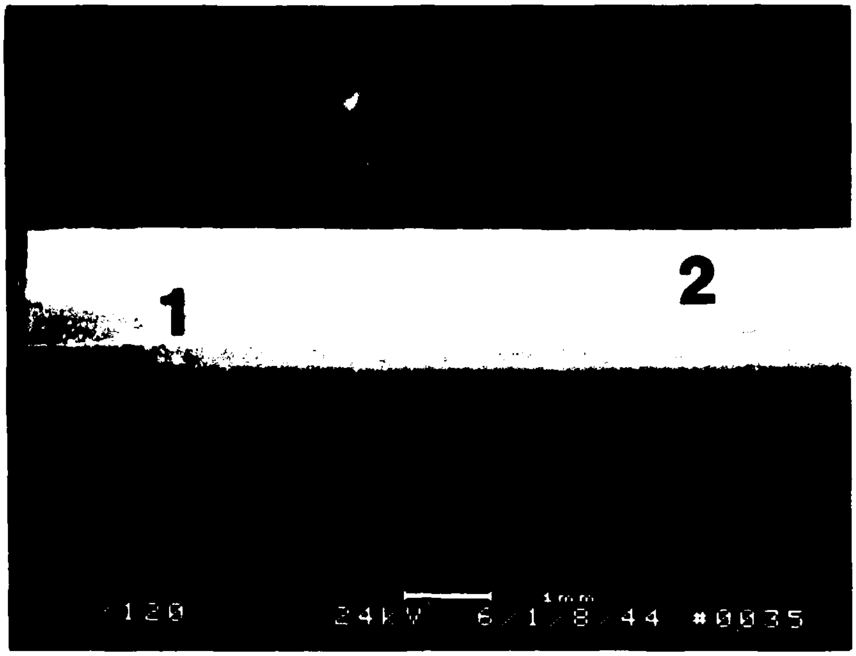


Figure 8. Scanning electron microscope photograph of impact area 4 of graphite epoxy sample No. 16-1-8. The cracks and delamination are clearly visible.

After impact tests were completed, these panels were ultrasonically inspected and no damage was observed. It is felt that the impact of the ice was spread over the flat surface of the ice pellet and that the ice was too soft to efficiently transfer its energy to the surface of the panel. Work should continue for simulated hailstone damage in a future program.

Task 4. Correlation of Dye Color to Impact Loads

During this task, the impact level was to be correlated to the intensity of the dye color or to different colors of indication. Some initial success was accomplished. This is illustrated in Figures 11 and 12. Figure 11 shows the effect of impact on a coating made from Run No. SP-63 plus neat CVL in the Air Force polyurethane-based paint coating. Both 7.5 and 10 ftlb impacts leave observable impacts, but this coating presently turns to a light-bluish color over a period of a few days. Figure 12 shows the impact-sensitive capsules in a silicon-based paint. This system shows indications for impact of 5 ftlbs and greater. In addition, the coating in this paint system does not change colors as it does in the Air Force polyurethane-based paint. To overcome the problem of gradual formation of bluish color on the panel, changes were made in the capsule formulation. Both the dye and the activator were encapsulated. The results of this encapsulation process prevented the activation from occurring and the coating did not turn bluish after several weeks. However, the intensity of the indication obtained from various levels of impact was considerably lowered. This result is shown in Figure 13.

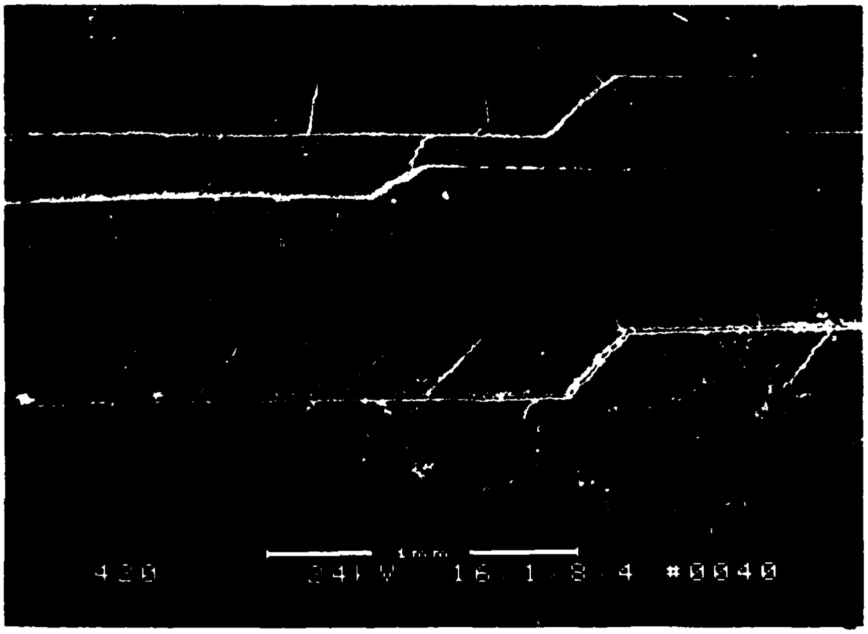


Figure 9. Magnified view of area 1 in scanning electron microscope photograph of impact area 4 of graphite epoxy sample No. 16-1-8.

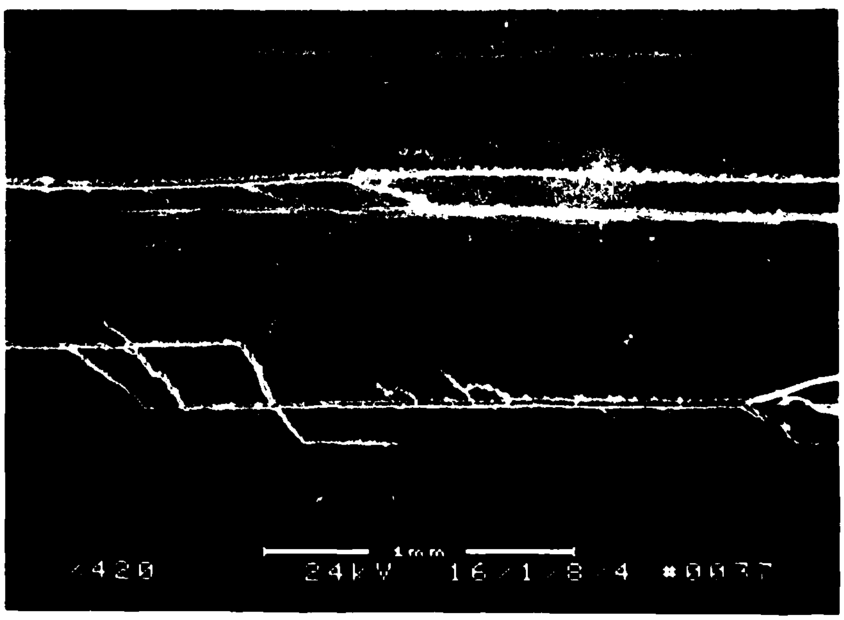


Figure 10. Magnified view of area 2 in the scanning electron microscope photograph of impact area 4 of graphite epoxy sample No. 16-1-8.

TABLE 6. PARAMETERS OF SIMULATED HAILSTONE IMPACT

Panel Number	Ice L (inch)	Ice M (grams)	Velocity (ft/sec)	Impact Level (ftlbs)
16-1-2	2 to 4	18 to 37	101 to 199	3 to 25
16-1-3	2 to 3	19 to 30	104 to 190	5 to 14
16-1-4	2 to 3	18 to 29	23 to 276	0.2 to 13
16-1-5	2 to 3	19 to 30	164 to 230	9 to 27
16-1-6	2 to 3	17 to 29	No valid data	
48-3-1	2 to 3	19 to 30	147 to 264	7 to 36
48-3-2	2 to 3	33 to 45	368 to 465	104 to 120
48-3-3	2 to 3	19 to 29	143 to 165	7 to 14
48-3-4	2 to 3	36 to 45	362 to 422	100 to 110
48-3-5	2 to 3	35 to 45	406 to 471	100 to 130
48-3-6	2 to 3	35 to 46	414 to 469	109 to 130

Task 5. Encapsulated Dye Development for Surface Impact Damage Indicator Systems

The purpose of this final task was to clearly demonstrate the capability of an encapsulated dye system to indicate the location and intensity range of impact damage to composite structures. This goal was not completed. The major problem seems to be associated with the chemical reactivity of the Air Force polyurethane-based paint.

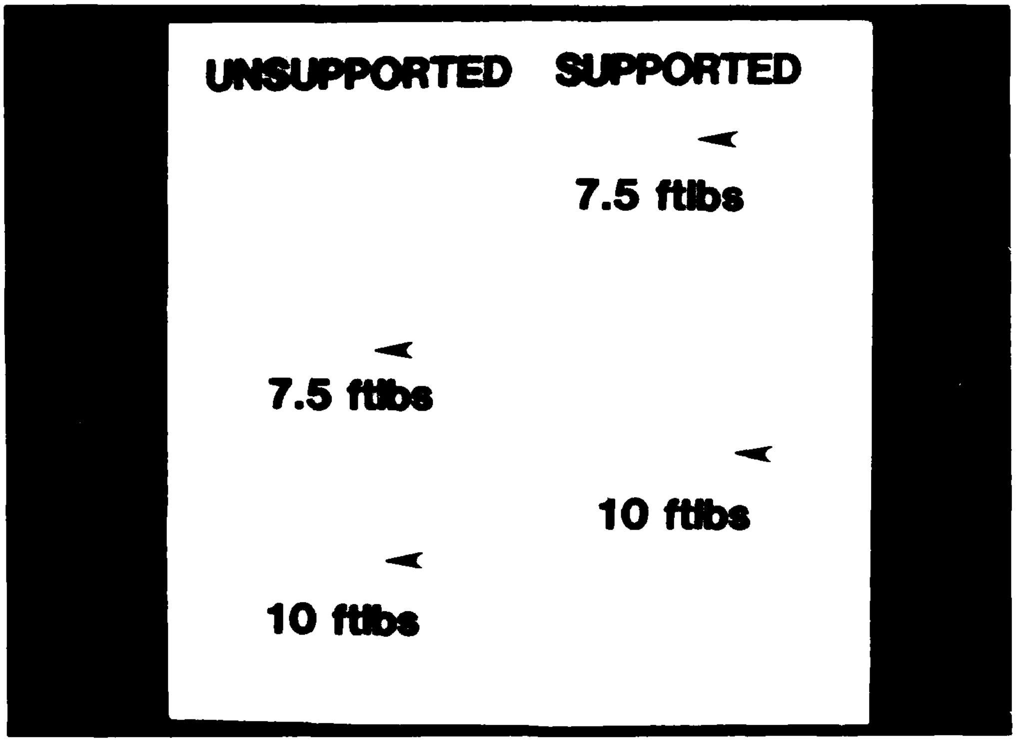


Figure 11. Photograph showing the effect of impact on a coating made from run No. WP-63 plus neat CVL in the Air Force polyurethanethane-based paint coating. Both 7.5 and 10 ftlb impacts leave observable indications, but this coating presently turns to a light-bluish color over a period of a few days.

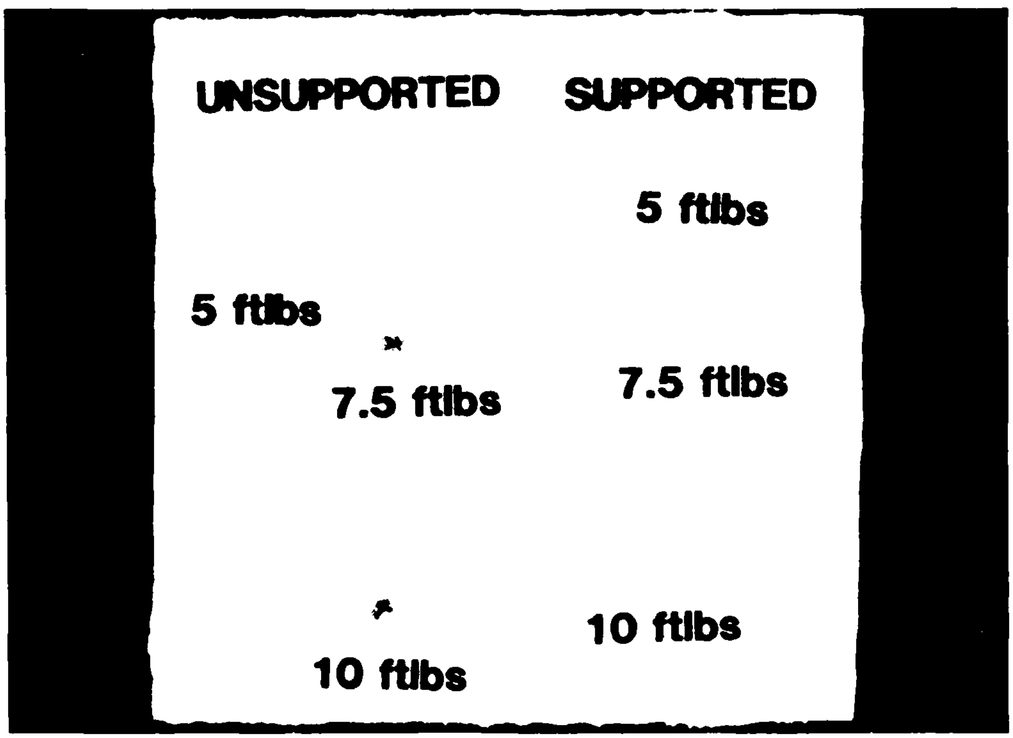


Figure 12. Photograph showing the impact-sensitive capsules in a silicon-based paint. This system shows indications for impacts of 5 ftlbs and greater.

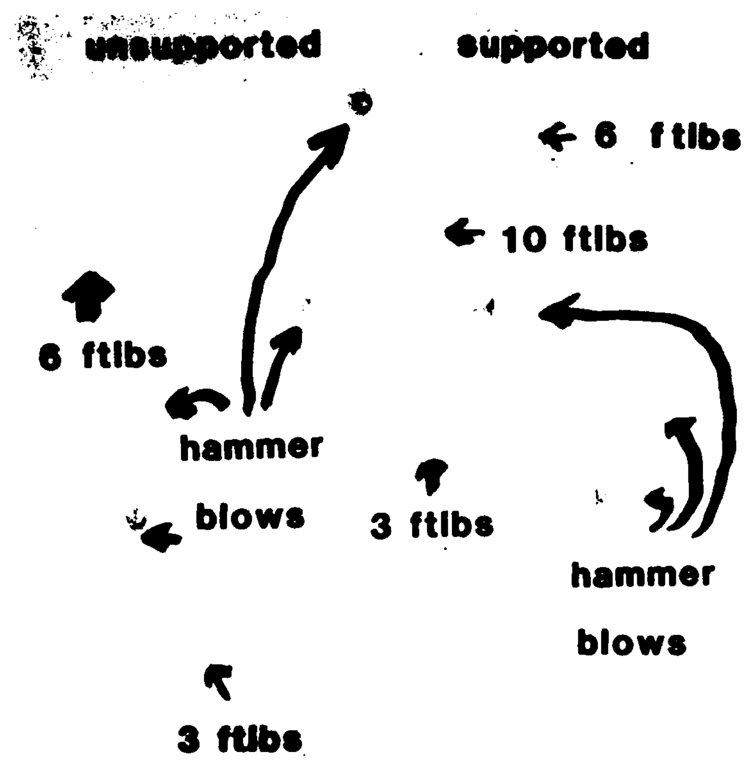


Figure 13. Photograph of composite panel with coating that has both the dye and developer encapsulated in separate capsules. The intensity of the impact is very low.

CONCLUSIONS

All the objectives of this program were met, with the exception of demonstrating the capability of developing an encapsulated coating that can indicate the range of impact as a function of color intensity or different colors. Our conclusions from this work are the following:

1. Impact-sensitive coatings based upon microencapsulation of dye and developer components can be developed that indicate impact.

2. Impact tests analyzed using destructive testing have shown that impacts in the range of 5 ftlbs can cause internal damage to the composite without causing any visible damage to the surface. In addition, through-transmission ultrasonic nondestructive evaluation (NDE) techniques can be used to detect this damage.

3. The impact coating indication can be correlated to the impact damage. The larger the area of coating indication, the greater the impact, as shown in Figure 12.

4. Impact-indicative coatings contain microcapsules that are small enough so that the coating can be sprayed onto a surface without clogging the spray nozzle or breaking the capsules.

RECOMMENDATIONS FOR FUTURE WORK

This program has clearly shown potential for the impact-sensitive coating concept. In order to make this a fieldable technique, the following recommendations for future work are suggested:

1. Continue the work to improve the sensitivity of the coatings which have both the dye and activator encapsulated in the coating. This will include fine-tuning the capsule size control, optimizing the payload level, improving capsule drying conditions, and further evaluating the solvent effects.

2. Develop different colored indicators (both blue and red were utilized during this work).

3. Continue the development of a UV fluorescent indicator system.

4. Conduct extended shelf life studies including the effects of temperature, humidity, and moisture.

Once these steps have been accomplished, it will be possible to correlate the level of impact indication with thdemonstrate the capability of an encapsulated dye system to indicate the location and intensity range of impact ddamage to composite structures. This goal was not completed. The major problem seems to be associated with the chemical reactivity of the Air Force polyurethane-based paint.

REFERENCES

1. Fourth Government/Industry Briefing on Damage Tolerance of Composites Program, San Antonio, Texas, May 22, 1986.

2. Development of Pressure-Sensitive Devices Employing Microcapsules, sponsored by Department of Health, Education, and Welfare, U.S. Public Health Service Hospital, 1969.

3. Development of Uniformity and Improved Shelf Life in Pressure-Sensitive Microcapsule System, sponsored by U.S. Public Health Service Hospital, 1971.

4. Microencapsulation of Phos-Chek P/30 Flame Retardant, sponsored by U.S. Army Natick Laboratories, 1981.

END

11-87

DTIC